

REMARKS/ARGUMENTS

Favorable consideration of this application, as presently amended and in light of the following discussion, is respectfully requested.

Claims 1 and 3 are presently pending in this application, Claim 1 having been amended and Claims 2 and 4 having been canceled by the present amendment.

In the outstanding Office Action, Claims 1-3 were rejected under 35 U.S.C. §102(b) as being anticipated by JP 10-024274 (hereinafter "JP '274"); and Claim 4 was rejected under 35 U.S.C. §103(a) as being unpatentable over JP '274.

Claim 1 has been amended herein. These amendments find support in the specification, claims and drawings as originally filed, for example, the specification, paragraphs 19-20, and no new matter is believed to be added thereby. If, however, the Examiner disagrees, the Examiner is invited to telephone the undersigned who will be happy to work in a joint effort to derive mutually satisfactory claim language.

Before addressing the rejection based on the cited reference, a brief review of Claim 1 as currently amended is believed to be helpful. Claim 1 is directed to a method for decomposing a plastic and recites "treating a thermosetting resin which comprises a polyester and its crosslinking moiety, with subcritical water of a temperature lower than the thermal decomposition temperature of the thermosetting resin; and recovering a compound comprising an acid residue derived from the polyester and a residue derived from the crosslinking moiety, obtained from the treating of the thermosetting resin, wherein the subcritical water contains an alkaline salt in an amount of not less than 2 molar equivalents relative to the theoretical number of the moles of an acid residue which is contained in a compound comprising the acid residue derived from the polyester and a residue derived from the crosslinking moiety, obtained by the decomposition of the thermosetting resin."

It is respectfully submitted that JP '274 is not believed to teach or suggest "recovering a compound comprising an acid residue derived from the polyester and a residue derived from the crosslinking moiety, obtained from the treating of the thermosetting resin, wherein the subcritical water contains an alkaline salt in an amount of not less than 2 molar equivalents relative to the theoretical number of the moles of an acid residue which is contained in a compound comprising the acid residue derived from the polyester and a residue derived from the crosslinking moiety, obtained by the decomposition of the thermosetting resin" as recited in amended Claim 1.

More specifically, JP '274 only describes a process for thermally decomposing (*i.e.*, C-C linkages are also decomposed) a thermosetting resin such as epoxy resins and phenolic resins with supercritical water to recover thermal decomposition products (phenol monomers) as it becomes apparent from the following discussions.

First, regarding the supercritical water, the Examples of Junya only describe a process for decomposing epoxy resins or phenolic resins with supercritical water at 400°C.¹ In hydrolysis, the value of the product of H⁺ ion and OH⁻ ion is important. The value of the ion product in subcritical water as used in the method of Claim 1 is about a thousand times as high as that in water at ordinary temperature. Accordingly, subcritical water has a very high activity for hydrolysis. In contrast, the value of the ion product in supercritical water is rapidly decreased at a temperature higher than the supercritical temperature (374.4°C),² and the value at 400°C is nine orders of magnitude less than that in subcritical water and about six orders of magnitude less than that in water at ordinary temperature. Namely, there are little

¹ See, JP '274, paragraphs 11-14.

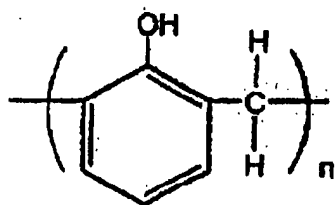
² See, the specification, paragraph 16.

ions required for hydrolysis in the reaction condition (supercritical water at 400°C) in the Examples of JP '274, *i.e.*, the thermal decomposition predominates in the reaction condition.³

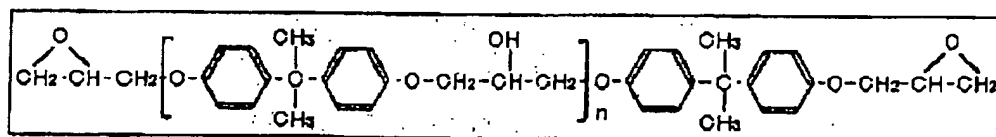
Second, regarding the thermal decomposition temperatures of phenolic resins and epoxy resins, the thermal decomposition temperature of phenolic resins is 300 to 350°C and the thermal decomposition temperature of epoxy resins is 200 to 250°C.⁴ Therefore, the temperature (400°C) of supercritical water in the Examples of JP '274 is higher than the thermal decomposition temperatures of phenolic resins and epoxy resins.

Third, regarding the decomposition product to be recovered, phenol monomers recovered in the Examples of JP '274 are thermal decomposition products of the thermosetting resin (phenolic resins and epoxy resins). Phenolic resins are phenol monomers linked together via methoxy groups, and epoxy resins have a chain of bisphenol A structures (see the following formulas). Therefore, if phenol monomers are obtained from these resins as decomposition products, it is considered that these resins are thermally decomposed (for example, the middle and both ends of bisphenol A structures in epoxy resins are decomposed).

Phenolic resins



Epoxy resins (bisphenol A type).



³ See, for example, the attached American Chemical Society Symposium Series, 518(1993)), page 38, Fig. 2.

⁴ See, for example, the attached Phenolic Resin Guidebook, p. 116 to 117.

In contrast, the method of Claim 1 is made by focusing on the structural feature (a polyester and its crosslinking moiety) of a thermosetting resin such as an unsaturated polyester (UP) resin, and is characterized by the efficient recovery of a crosslinking moiety-containing molecule such as a styrene-fumaric acid copolymer (SFC) (hereinafter "SFCs"). The SFCs have a high potential as a functional polymer and are reusable as a high value-added material such as a low shrinking agent, a surfactant, a dispersant for pigment, etc. The JP '274 process, on the other hand, is directed to the thermosetting resin having a matrix structure obtained by crosslinking a part of the main chains containing monomer units such as phenol and bisphenol A linked together via methoxy linkages, via ester linkages. The structure of the thermosetting resin to be decomposed in the JP '274 process is clearly different from that of the thermosetting resin (*e.g.*, UP resin) comprising a polyester and its crosslinking moiety to be decomposed in the method of Claim 1. As described above, the main reaction in the reaction condition of JP '274 is thermal decomposition rather than hydrolysis. Therefore, phenol monomers derived from the main chain containing monomer units such as phenol and bisphenol A via methoxy linkages, are only obtained in the JP '274 process. Even if the ester linkages of the thermosetting resin are hydrolyzed in the JP '274 process, the resin essentially have no molecular structure having a high potential as a functional polymer containing many carboxylic acid functional groups, and therefore, a high-value added material such as SFC cannot be recovered in the JP '274 process.

Also, in the method of Claim 1, subcritical water which mainly hydrolyzes ester linkages is used, and the decomposition of the thermosetting resin is carried out at a temperature lower than the thermal decomposition temperature of the resin such that the crosslinking moieties of the resin are not decomposed (*i.e.*, C-C linkages are not decomposed), for the efficient recovery of a functional polymer such as SFC. In addition, a given amount of alkaline salt is also used in the method of Claim 1 for this purpose. On the

other hand, in the JP '274 process, the concentration of NaOH in water is preferably 2%, more preferably 0.2% in terms of the effects of the addition, post-treatments, etc.⁵ In the Examples of JP '274, the recovery percentage of the resultant phenol monomers was 8% when only water was used,⁶ and 10% when NaOH: 500 g (corresponding to a concentration of 1%) was added to water.⁷ There are little effects of the addition of NaOH in terms of the recovery percentage of phenol monomers.

This is due to that the main reaction in the Examples of JP '274 is thermal decomposition (*i.e.*, C-C linkages are also decomposed) rather than hydrolysis of esters, as described above, and therefore, the addition of NaOH, which can accelerate hydrolysis of esters, is almost meaningless. In contrast, for the efficient recovery of SFCs, an alkaline salt is used in an amount of not less than 2 molar equivalents relative to the theoretical number of the moles of an acid residue which is contained in SFCs in the method of Claim 1. The amount is much higher than the amount required for the acceleration of hydrolysis of esters. By using the above amount of an alkaline salt, the efficient recovery of desired SFCs is attained.

To illustrate this point, submitted herewith is the Declaration of Mr. Nakagawa demonstrating experiments and their results. In the Experiments, the effects of the addition of KOH were confirmed when an unsaturated polyester (UP) (free of styrene and uncrosslinked) was decomposed with subcritical water. The decomposition condition was the same as that in the Examples of the specification.

In the Experiment 1 (free of KOH, water alone), the decomposition percentage of UP was 100%, the recovery percentage of glycol was 59%, and the recovery percentage of organic acid was 85%. The mean value of recovery percentages was 72%, since the glycol

⁵ See, JP '274, paragraph 9.

⁶ See *id.*, Example 1, paragraph 11.

⁷ See *id.*, Example 2, paragraph 12.

and the organic acid were contained in an equivalent amount in UP. When KOH was used in an amount of 1.9 molar equivalents relative to the theoretical number of moles of acid residue in SFCs (Experiment 2, *1 in Table A), the decomposition percentage was 100%, the recovery percentage of glycol was 86%, and the recovery percentage of organic acid was 62%. The mean value of recovery percentages was 74%, which is the same as that in the Experiment 1 (water alone). The same results were obtained when higher amounts of KOH were used (Experiments 3 and 4).

In the Experiments, there are little effects of the addition of KOH on the decomposition of uncrosslinked UP with subcritical water. In hydrolysis of polyesters, an alkaline salt such as KOH is generally used to provide water with OH^- ions in order to accelerate the reaction. However, there would be enough OH^- ions for hydrolysis of esters in subcritical water in the reaction conditions in the Experiments, even if the OH^- ions were not provided from KOH, since the value of the ion product in subcritical water is about a thousand times as high as that of water at ordinary temperature. Therefore, the addition of alkaline salt is almost meaningless in the decomposition of uncrosslinked UP.

Unlike when uncrosslinked UP is decomposed, the effects of the addition of alkaline salt in a given amount is pronounced when a thermosetting resin comprising a polyester and its crosslinking moiety is decomposed in the present invention.

In the Example 1 of the specification (water alone), the decomposition percentage of the thermosetting resin was 5.1%, the SFC formation percentage was 1%, and the glycol recovery percentage was 47%. When KOH was used in an amount of 1.9 molar equivalents relative to the theoretical number of moles of acid residue in SFC (Example 2), the decomposition percentage was 36%, and the glycol recovery percentage was 41%. However, the SFC formation percentage was still 1%. In contrast, when KOH was used in an amount of not less than 2 molar equivalents, 4.8 molar equivalents, (Example 3), the decomposition

percentage was increased to 87%, the glycol recovery percentage was increased to 72%, and the SFC formation percentage was drastically increased to 70%.

It is apparent that the main reaction in the Examples of the specification is hydrolysis of ester linkages in the polyester main chains of the thermosetting resin in view of the products (glycol, organic acid, and SFC).

The above Experiments demonstrate that the addition of KOH is almost meaningless in the decomposition of uncrosslinked UP with subcritical water, and thus it is apparent that the acceleration of hydrolysis of polyester by the addition of KOH is not the reason why the addition of KOH leads to the drastic increased SFC formation percentage in the decomposition of crosslinked UP with subcritical water.

Applicants respectfully submit that the result is caused by the improvement of the solubility of SFC with KOH. SFC in carboxylic acid form is insoluble in water, but K salt thereof (SFC-K) is soluble in water. In view of the glycol recovery percentage, it is apparent that about half of ester linkages of polyester main chains in the thermosetting resin were decomposed when KOH was used in an amount of 1.9 molar equivalents (Example 2). Despite this, the decomposition percentage and the SFC formation percentage were very low in Example 2. It is believed that SFC cannot dissolve into water, and remains along with inorganic materials in the bulk of the thermosetting resin mold, namely, when the resin on the surface of the bulk of the mold is decomposed, the resultant glycol dissolves into water, but insoluble SFC remains on the surface of the bulk, and prevents water from accessing the inside of the resin.

In contrast, when KOH is used in an amount of not less than 2 molar equivalents, SFC-K dissolves into water, the surface of the bulk of the thermosetting resin mold appears one after another, fresh water can always access the resin, the size of the mold rapidly decreases, and the ratio of water amount to the surface area of the bulk increases at an

accelerated rate. Namely, the Shrinking-Core model is developed, and the reaction is efficiently progressed.

Therefore, the effects of the addition of a given amount of an alkaline salt in the method of Claim 1 would not be obvious. In addition, thermosetting resins such as UP resin are generally used as a composite with inorganic materials, etc. Thus, if SFCs are present as an insoluble solid, it is difficult to separate SFCs from inorganic material etc. When a thermosetting resin comprising a polyester and its crosslinking moiety is decomposed with subcritical water containing an alkaline salt in an amount of not less than 2 molar equivalents, SFCs can be dissolved into water in the form of an alkaline metal, salt, and therefore, can be easily separated from inorganic materials. The method of Claim 1 according to the present invention provides such an advantage.

Based on the foregoing discussions, it is respectfully submitted that the subject matter recited in amended Claim 1 is believed to be distinguishable from JP '274, and because JP '274 fails to disclose the treatment and/or recovery as recited in amended Claim 1, its teachings are not believed to render the method recited in Claim 1 obvious.

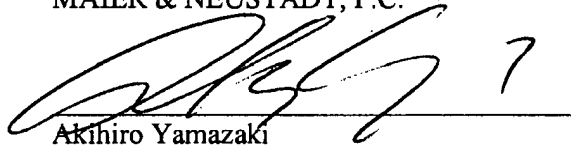
For the foregoing reasons, Claim 1 is believed to be allowable. Furthermore, since Claim 3 depend directly from Claim 1, substantially the same arguments set forth above also apply to this dependent claim. Hence, Claim 3 is believed to be allowable as well.

Application No. 10/593,081
Reply to Office Action of December 17, 2008

In view of the amendments and discussions presented above, Applicants respectfully submit that the present application is in condition for allowance, and an early action favorable to that effect is earnestly solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,
MAIER & NEUSTADT, P.C.

A handwritten signature in black ink, appearing to read 'Akihiro Yamazaki', is written over a horizontal line. To the right of the signature is a handwritten number '7'.

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Chapter 3

Supercritical Water Oxidation Technology
Process Development and Fundamental Research

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Hazardous organic wastes, including chlorinated hydrocarbons, in aqueous media containing salts can be effectively oxidized by treatment above the critical point of pure water (374°C, 221 bar). High destruction efficiencies may be achieved at low reactor residence times (approximately 1 minute or less) for temperatures above 550°C. Under these conditions, no NO_x compounds are produced. The high solubility of organics and oxygen and the low solubility of salts in supercritical water make it an attractive medium for both oxidation and salt separation. This paper reviews critical technology components and operations required for commercial-scale development of supercritical water oxidation (SCWO). These include fluid handling and compression, heat exchange and recuperative heat recovery, reactor and salt separator design and materials considerations. In addition, a summary of fundamental research activities in the areas of oxidation reaction kinetics and mechanisms and solid salt nucleation and deposition is presented in the context of its impact on SCWO process development.

Oxidation carried out in a supercritical water environment at temperatures above 374°C and pressures above 221 bar (22.1 MPa) provides a viable method for the efficient destruction of organic wastes in a fully contained system. This patented process has been termed supercritical water oxidation, SCWO, and has been under commercial development for about the past 11 years by MODAR, Inc. (1-7). The process has also proven suitable for treatment of human metabolic wastes, and is being considered for use in life support systems on long-term spaceflights (8-11). A relatively recent variation of SCWO involves carrying out the process in a deep well utilizing the hydrostatic head of fluid in the wellbore to help provide the necessary pressure level (see, e.g., Gloyna (12)).

The main objectives of this paper are twofold: (1) to review the status of above-ground SCWO technology from a process engineering perspective, and (2) to review fundamentally focused research on reaction kinetics, phase equilibria and solid salt separation critical to understanding the chemistry and phase behavior of reactions carried out in the SCWO process. The effectiveness of SCWO for treating a wide range of waste stream compositions will be reviewed. The status of development of SCWO as a commercial-scale process along with a discussion of remaining technical issues and critical sub-process elements will also be presented.

is not required. Nonetheless, solids removal and storage are necessary, and water effluent polishing units incorporating ion exchange may be needed to remove small concentrations of dissolved metal ions. Major drawbacks of SCWO are the high pressures required (>230 bar) and the fact that corrosion at certain points in the process is significant. For some wastes, solids handling can also pose difficulties.

Properties of Supercritical Water

In the region near the critical point, the density of water (shown in Figure 1) changes rapidly with both temperature and pressure, and is intermediate between that of liquid water (1 g/cm^3) and low-pressure water vapor ($<0.001 \text{ g/cm}^3$). At typical SCWO conditions, the water density is approximately 0.1 g/cm^3 . Consequently, the properties of supercritical water are quite different from those of liquid water at ambient conditions, particularly those properties related to solvation.

Insight into the solvation characteristics and molecular structure of the aqueous system can be obtained by experimental measurements of dielectric constant, ionic dissociation constant, and Raman spectral emissions in passing from the subcritical to supercritical regions. Figure 2 shows that the static dielectric constant of water at 25 MPa drops from a room-temperature value of around 80 to about 5 to 10 in the near-critical region, and finally to around 2 at 450°C and above (21-23). Along the same isobar, as shown in Figure 2, the ionic dissociation constant falls from 10^{-14} at room temperature to 10^{-18} in the near-critical regime and to 10^{-23} under supercritical conditions (24). Furthermore, Raman spectra of deuterated water in the supercritical region show only a small residual amount of hydrogen bonding (25,26). As a result, supercritical water acts as a non-polar dense gas, and its solvation properties resemble those of a low-polarity organic. Hydrocarbons exhibit generally high solubility in supercritical water.

Near the critical point, the solubility of an organic compound in water correlates strongly with density and is thus very pressure dependent in this region. Benzene solubility in water is a good example (27,28). At 25°C , benzene is sparingly soluble in water (0.07 wt%). At 260°C , the solubility is about 7 to 8 wt% and fairly independent of pressure. At 287°C , the solubility is somewhat pressure dependent, with a maximum of 18 wt% at 20 to 25 MPa. In this pressure range, the solubility rises to 35 wt% at 295°C , and at 300°C , the critical point of the benzene-water mixture is surpassed. When the mixture becomes supercritical, by definition, there is only a single phase. Thus, the components are miscible in all proportions.

Other hydrocarbons exhibit similar solubility behavior near 25 MPa: Binary mixtures of *n*-alkanes ($\text{C}_2\text{--C}_7$) and water become supercritical (and, therefore, completely miscible) at temperatures below about 370°C (27,29,30). Supercritical water also shows complete miscibility with "permanent" gases such as nitrogen (31), oxygen and air (32), hydrogen (33), and carbon dioxide (34,35), in addition to small organics like methane (36). The loci of critical points of the binary mixtures containing water have also been reported by Franck and co-workers for the aforementioned organic substances and gases. Alwani and Schneider (37) have reported the phase behavior of aromatic hydrocarbons (including 1,3,5-trimethylbenzene and naphthalene) with supercritical water. All of the compounds studied to date are completely miscible with water above 400°C at 25 MPa. Franck and his research group have also extended earlier phase behavior information for binary systems to include phase equilibrium data for ternary systems containing salts such as NaCl, CaCl_2 , and NaBr (36,38,39).

In contrast to the high solubility of organics, the solubility of inorganic salts in supercritical water, as shown in Figure 3, is very low. Many salts that have high solubilities in liquid water have extremely low solubilities in supercritical water. For example, NaCl solubility is about 37 wt% at 300°C and about 120 ppm at 550°C and 25 MPa (44); CaCl_2 has a maximum solubility of 70 wt% at subcritical temperatures,

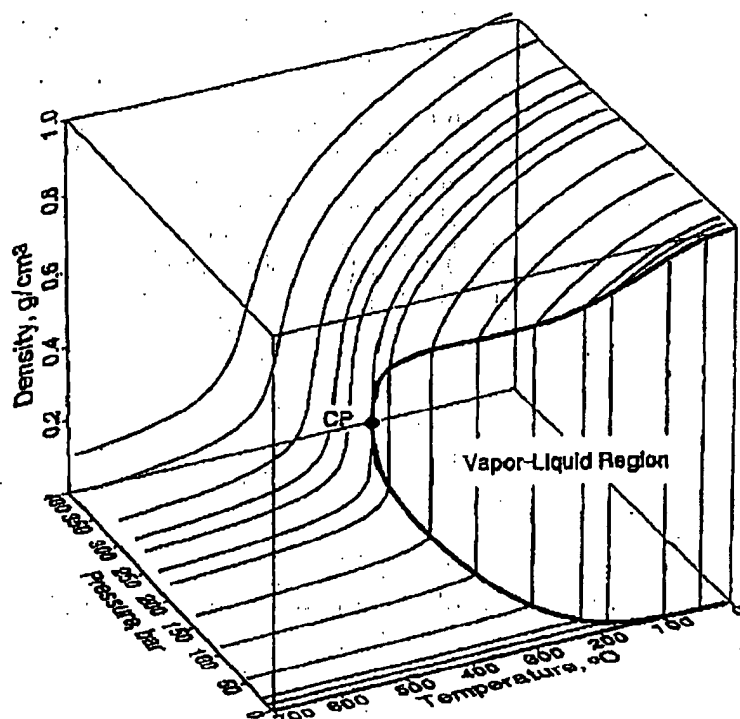


Figure 1. Pressure-Temperature-Density Behavior of Pure Water. CP denotes the critical point; the vapor-liquid region indicates conditions under which two phases are present. Density calculated from the equation of state of Haar *et al.* (20).

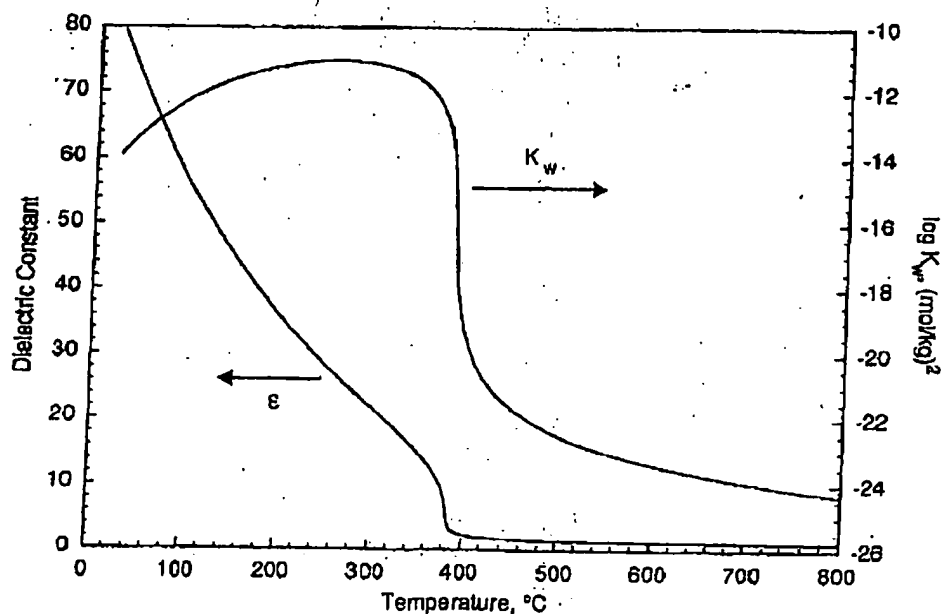


Figure 2. Solvation Properties of Pure Water at 25 MPa. Properties shown are the dielectric constant ϵ (21) and the ionic dissociation constant K_w (24).

Attachment 2

Partial English translation of Polymeric Mechanical
Materials - Phenolic Resin Guidebook, pages 116 to 117

(p.116)

Table 2.1 Organic Polymers Having Superior Heat Resistance
(omitted)

C. Heat resistance of Phenolic Resins

a. Heat resistance of Phenolic Resins themselves

Fig. 2.1 ⁵⁾ shows the heat resistance of phenolic resins compared to other resins. This graph shows a decomposition amount of each resin by thermolysis in vacuum.

(omitted)

Fig. 2.1 Relative Thermal Stability Of Plastics

From the above graph, it is apparent that phenolic resins have the second largest thermal stability after silicones. When these resins are heated in air, the temperatures where the weights of the resins are rapidly reduced by thermolysis are as follows ⁶⁾:

(p.117)

Silicones	350 to 400°C
Phenolic resins	300 to 400°C
Epoxy resins	200 to 250°C
Polyester	150 to 200°C

In this case, silicones show the largest thermal stability, followed by phenolic resins. Fig. 2.2 ⁷⁾ shows the weight reducing of phenolic resins during heating in air.

(omitted)

**Fig. 2.2 Weight Reducing Of Phenolic Resins During Heating
In Air**

(omitted)

Polymeric Mechanical Materials - Phenolic Resin Guidebook

Published on October 1, 1967.

Editor	The Committee for Polymeric Mechanical Materials of The Society of Polymer Science, Japan
Publisher	Hatsugoro Nanjyo
Published by	Kyoritsu Shuppan Co., Ltd., 4-19-6 Kohinata, Bukyo-ku, Tokyo

表 2.1 すぐれた耐熱性をもつ有機高分子

名 称	構 造	耐熱性の特長
ポリフェニル (ベンゼンポリマー)	$\left[\text{C}_6\text{H}_4 \right]_n$	融点 550°C 以上
ポリチアゾール	$\left[\text{C}_6\text{H}_4 - \text{N} = \text{N} - \text{S} - \text{C}_6\text{H}_4 \right]_n$	500°C 以上に十分に耐える耐熱性をもつ
ポリイミド	$\left[\text{N} - \text{C}(=\text{O}) - \text{C}_6\text{H}_4 - \text{C}(=\text{O}) - \text{N} - \text{R} \right]_n$	220°C, 1,300 時間加熱で 5.6% の重量減少を示すのみ
ポリベンツイミダゾール	$\left[\text{C}_6\text{H}_4 - \text{N} = \text{N} - \text{C}_6\text{H}_4 - \text{N} = \text{N} - \text{C}_6\text{H}_4 \right]_n$	熱天びんで測定した 550°C の加熱減量は 5% 程度, 770°C 以下では熔融せず, 現在最高の耐熱性をもつ高分子

C. フェノール樹脂の耐熱性

a. フェノール樹脂自身の耐熱性

フェノール樹脂が他の樹脂と比較してどの程度の耐熱性があるかを図 2.1⁵⁾ に示した。

この図は各種樹脂の真空中での加熱分解量を示したものであるが、これによるとフェノー

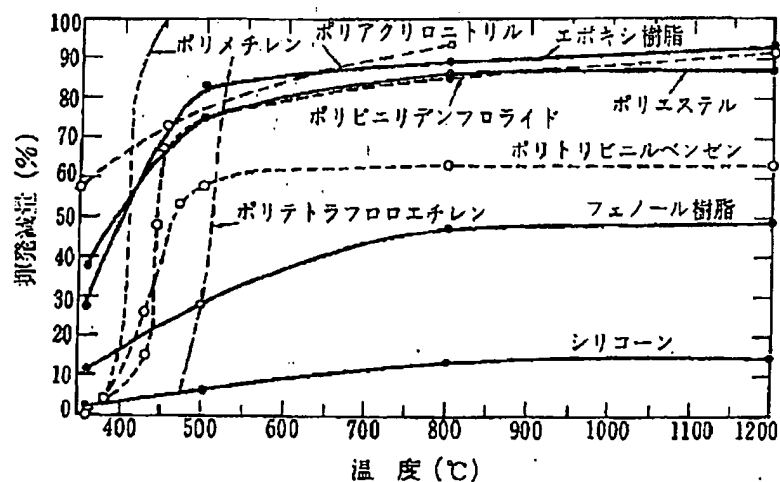


図 2.1 プラスチックの相対的熱安定性

ル樹脂はシリコーンに次いで熱安定性のよいことがわかる。また、空気中で加熱した場合、急速に熱分解し重量が減少する温度は

シリコーン	350~400°C
フェノール樹脂	300~350°C
エポキシ樹脂	200~250°C
ポリエステル	150~200°C

であり⁹⁾、この場合にもシリコーンが最もよい耐熱性を示し、フェノール樹脂がこれに次いでいる。図 2.2⁷⁾ はフェノール樹脂を空气中で加熱したときの減量を示した。

一般に、共役二重結合を多く含むポリマーは耐熱性がすぐれており、また、鎖中にメチレン結合が存在すると熱安定性が低下する。そして、これをフェノール樹脂が示す耐熱性と関連づけて解釈すると、フェノール樹脂の熱安定性は

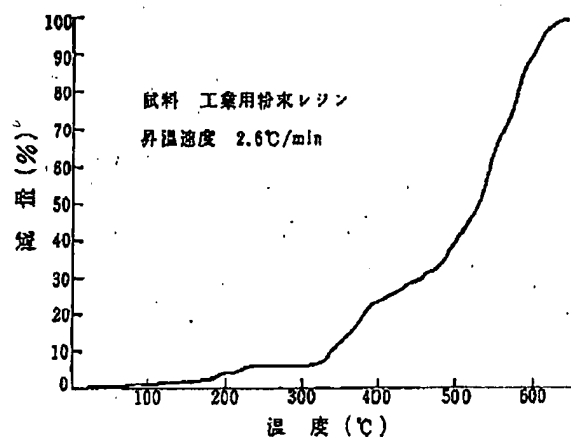


図 2.2 フェノール樹脂の空气中における加熱減量

① 網目構造中のメチレン橋の高度の安定性

② ベンジル基の共鳴安定性

③ 網目構造中のベンゼン核の高密度、すなわち共役二重結合の高密度の存在のためと考えられる⁹⁾。

また、フェノール樹脂の加工性をよくするためにアルキルフェノールが使用されることは周知のとおりであるが、アルキル化フェノール樹脂の熱安定性については、次のような一般的結論がくだされている⁹⁾。

① アルキル基の置換は熱安定性をわるくする

② ある鎖長までは置換基の長いほど安定性はわるい

③ アルキル基の分枝は安定性をわるくする

このことから他の樹脂と同様に、フェノール樹脂の場合にも、耐熱性高分子材料として加工性を付加することはむずかしく、Marvel の要求する条件に矛盾があり、彼自身もその矛盾は今後に課せられた問題であるとしている。

b. 基材と組み合わせたフェノール樹脂の耐熱性

高分子機械材料
フェニール樹脂
ガイナック

定価 2000 円



© 1967

昭和42年10月1日 初版1刷発行

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機械材料委員会

発行者 南條初五郎
東京都文京区小日向 4-19-6

印刷者 福田三郎
東京都渋谷区猿樂町 51

発行所 東京都文京区小日向 4-19-6
電話東京 (947) 2511 番 (代表)
振替口座 東京 57035 番
共立出版株式会社

NDC 578.482

共立製本所 印刷・製本

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